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Thickness dependent structure of β-$FeSi_2$ grown on silicon by solid phase epitaxy

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Abstract

Semiconducting β-$FeSi_2$ was grown on Si(0 0 1) substrates by depositing Fe layers of thickness 2, 4 and 6 nm at room temperature in an ultra-high vacuum system and subsequent in situ annealing at 600 °C for 15 min. The phase of the grown polycrystalline β-$FeSi_2$ was confirmed by electron diffraction and infrared reflectance measurements. Transmission electron microscopy analysis has shown that the structure of the formed β-$FeSi_2$ is thickness dependent. For an Fe layer of thickness 2 nm, β-$FeSi_2$ nanodots with a mean diameter of about 15 nm were grown, together with islands of irregular shape and linear or S-type arrays of β-$FeSi_2$ nanodots. For thicker Fe layers, the grown β-$FeSi_2$ consisted of continuous polycrystalline layers.
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Keywords: Solid phase epitaxy; β-$FeSi_2$; Nanodots; Polycrystalline layer

1. Introduction

The preparation of artificial low dimensional structures for electron confinement is one of the most challenging research fields of the solid-state technology [1]. The research of the quantum dots has increased dramatically over the past few years due to the predicted drastic improvement in the performance of optoelectronic devices. Phenomena of self-assembly have been observed, besides the compound semiconductors of group IV, in a wide range of material and substrate combinations [2]. Heteroepitaxial growth of strained semiconductor structures have attracted great interest recently, owing to their scientific interest and possible technological importance as quantum dots [3]. The generated dots, through the combination of growth kinetics and strain effects, show a rather narrow size distribution on the substrate.

The semiconducting iron disilicide (β-$FeSi_2$) has attracted much attention due its potential application in silicon-based optoelectronics. The band gap of β-$FeSi_2$ is direct (about 0.87 eV at room temperature) [4], making this material a potential candidate for infrared light emitting devices or detectors, whereas a lot of effort has been made to prepare semiconducting polycrystalline β-$FeSi_2$ layers [4–7], less work has been performed to grow β-$FeSi_2$ nanodots on Si substrate.

Earlier, few publications reported island or wire-like aggregation of silicides on Si substrates. $TiSi_2$ islands were observed on both Si(1 0 0) and Si(1 1 1) substrates by depositing Ti layers at elevated temperatures, followed by high temperature annealing [8]. Nanostructures of $CoSi_2$ were prepared on Si(1 0 0) substrate by reactive deposition epitaxy (RDE) and their nucleation and evolution were studied during the annealing [9]. Formation of the rare earth Er, Dy, Ho silicide nanowires have also been reported [10,11]. Recently, β-$FeSi_2$ islands were grown by means of Fe^+ implantation into Si(1 0 0), followed by thermal annealing at 800 °C [12]. In this work, β-$FeSi_2$ was grown on Si(0 0 1) substrates by solid phase epitaxy, with variable thickness of the Fe layer. The phase of the formed silicide was identified by infrared (IR) reflection measurements,

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whereas the microstructure of the silicide is characterized by transmission electron microscopy (TEM).

2. Experimental

Single crystal n-type (0 0 1) Si wafers were used as substrates for the growth of β-$FeSi_2$. First, the substrates were cleaned by a standard chemical process and then dipped in HF solution before loading them into the oil free evaporation chamber. After evacuation down to about 7.5×10^{-9} Torr and prior to the Fe evaporation, the Si substrates were annealed in situ for 5 min at 800 °C to remove the native oxide. At the base pressure of about 2×10^{-8} Torr, high-purity Fe layers (99.9%) of thickness 2, 4, and 6 nm were deposited by e-gun evaporation on the Si substrates with a deposition rate of 0.01 nm/s. During deposition, the substrate temperature was maintained at room temperature. Immediately after the Fe deposition, the specimens were annealed in situ at 600 °C for 15 min in the vacuum chamber to produce the iron silicide. As a reference sample for the IR measurements, we used bulk polycrystalline β-$FeSi_2$, prepared by sintering pure elements of iron and silicon in the atomic ratio 1:2 under inert argon atmosphere. Details for the growth of the bulk β-$FeSi_2$ sample are presented elsewhere [4].

IR measurements were carried out with a Bruker IFS 113v spectrophotometer, operated under vacuum. The spectra of the samples were collected at room temperature in specular reflectance and transmittance modes in the FIR region of 900–120 cm^{-1} with unpolarized and polarized light. The resolution was 4 cm^{-1} and for each spectrum 128 consecutive scans were recorded. A gold mirror was used as a reference sample. A JEOL-100 CX electron microscope operated at 100 kV was used to investigate the structure of the silicides formed on the silicon substrates after thermal treatment. Two types of specimens were prepared for each sample: one having the silicon–silicide interface perpendicular to the electron beam (planar view) and the other parallel to the electron beam (cross-sectional view). Both cases were considered necessary to obtain information on the morphology of the silicide films.

3. Results and discussion

Fig. 1 shows typical reflectivity spectra of the silicide layers formed by depositing Fe layers of thickness 2, 4 and 6 nm, together with the spectrum of the bulk β-$FeSi_2$ polycrystalline crystal, obtained with unpolarized light. In the FIR region, the spectra exhibit intensive phonon structure, presenting a dominant characteristic pattern with the contribution of five phonons. The reflectivity spectra of the grown silicide layers show the vibrational structure of the β-$FeSi_2$ phase, which is deduced from comparison with the reflectivity spectrum of the bulk β-$FeSi_2$ sample. From Fig. 1, it is clearly shown that the reflectivity spectra become increasingly more structured with decreasing layer thickness, leading to the conclusion that the degree of crystallinity increases as the film thickness decreases. In order to identify a possible anisotropy of the silicide layers, we have obtained detailed polarized spectra from all samples (not shown). No clear anisotropy was noticed in the IR measurements, attributed to the spread of the crystallite domain orientation in the silicide film.

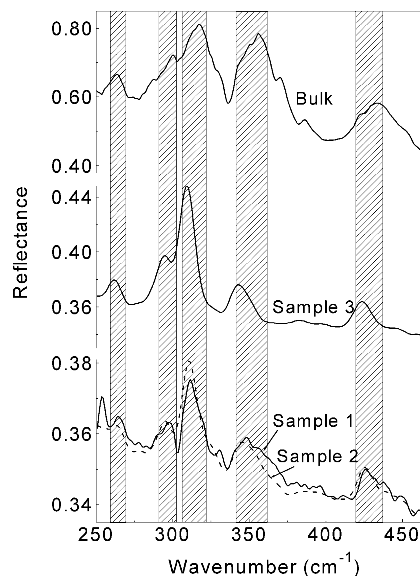

Fig. 1. IR reflectivity spectra of the three silicide layers formed by depositing Fe layers of thickness 2 nm (sample 1), 4 nm (sample 2) and 6 nm (sample 3) and annealing at 600 °C for 15 min in vacuum. For comparison, the spectrum obtained from bulk β-$FeSi_2$ polycrystalline crystal is also presented.

In the reflectivity spectra of Fig. 1, additional peaks can also be identified. The origin of these peaks might be attributed to the contribution from different oriented structures of the film. From the IR study of single-crystalline β-$FeSi_2$ specimens with polarized light [13], it has been demonstrated that there are remarkable differences between the IR spectra obtained at different polarizations with respect the crystal axis, with the position of the peaks and their relative intensity being different. Consequently, in our polycrystalline β-$FeSi_2$ films, the observed simple five phonon pattern can be understood as the convolution of individual contributions from crystal grains having different orientations and sizes.

Fig. 2(a) and (b) show the planar-view bright field TEM images, obtained from two different areas of the specimen prepared by depositing 2 nm thick Fe film on silicon at room temperature, followed by in situ annealing at 600 °C for 15 min. It is clearly seen that the Si substrate is covered by the β-$FeSi_2$ silicide grown in different shapes. In some areas of the specimen, in addition to the linear or S-type arrays of β-$FeSi_2$ nanodots with diameter about 8 nm, β-$FeSi_2$ grains in the form of coalesced islands with irregular shape are formed on the Si substrate as shown in Fig. 2(a). The diffraction pattern shows dotted rings as shown in the inset of Fig. 2(a), indicating the polycrystalline nature of the β-$FeSi_2$ material.

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